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<b>14. ABSTRACT</b>  This report results from a contract tasking Ioffe Physico-Technical Institute as follows: As detailed in an on-line proposal, the contractor will: 1) develop and build an optical device, fitted to a Fabry-Perot interferometer, to perform high-resolution quasielectric light scattering spectroscopy; 2) develop related techniques for registering quasielectric laser light scattering spectra; 3) measure temperature-dependent (77-300 degrees Kelvin) electrical conductivity to characterize metal/polymer/metal systems; 4) perform high-resolution Raman scattering mapping of epoxy/Al and teflon/Al interfaces to find quasaielectric scattering and the evolution of structural changes; 5) permorm Brillouin light-scattering measurements on epoxy/Al and teflon/Al interfaces for evolution of the longitudinal modulus from the surface ofpolymer close to polymer/Al interfaces; 6) perform Rayleigh and quasielastic light scattering measurements on epoxy/Al, epoxy/Ag, and teflon/Al interfaces to find the evolution of the relaxation time from the surface of polymer close to polymer/metal interfaces.					
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## SUMMARY OF FINAL REPORT

Project # 2007p

### 1. Title of the Project / Final Report

Development of High Spectral Resolution Technique for Registration Quasielastic Light Scattering Spectra Including Rayleigh and Brillouin Scattering as a Diagnostic Tool in Materials Characterization

For 3 years

### 2. Contracting Institute

Ioffe Physico-Technical Institute  
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### 3. Participating Institutes

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### 5. Commencement Date, Duration

1 June 2001, 3 years

### 6. Objectives of the Project

Promoting integration of scientists of CIS states into the international scientific community;

The main aim of the project is to carry out basic research on development high spectral resolution technique for registration quasielastic laser light scattering spectra.

### 7. Scope of Work and Technical Approach

In the 3 years of it's operation, the project has proven very successful in its mission to develop technique of Quasielastic Light Scattering Spectra Including

Rayleigh and Brillouin Scattering necessary as a Diagnostic Tool in Materials Characterization.

We have developed high-resolution technique for detection of quasielastic light scattering spectra based on the use of a scanning by gas pressure Fabry-Perot interferometer coupled to the double grating monochromator.

#### 8a. Technical Progress

We have designed and built new special metallic box with two optical windows for the Fabry-Perot interferometer and fine valve. High optical-quality and plane windows were made by us from the quartz glass. This system will allow performing photoelectric recording of the spectra together with high temperature and pressure stabilization.

Development of such a technique has allowed to demonstrate the possibility of detection of the interferometric high-resolution spectra of inelastic light scattering by longitudinal optical phonons in semi-insulating gallium phosphide crystals in the temperature range 7-300K.

For study optical properties of metal/polymer/metal systems and to reveal electrical characteristics of these materials we have measured the temperature dependent electrical conductivity (in the temperature range of 77-300 K) of metal/polymer/metal systems for determination mobility and concentration of carriers. In these system we planed to study the quasielastic and inelastic light scattering.

Our first study of electrical properties of the system with polymer-metal-polymer interfaces indicate the Ohm-law character of the conductivity for the polymer film inserted between metallic plates. Current-voltage characteristics measured in the wide temperature and potential ranges indicate that the temperature dependence of the conductivity is described by the exponential law, that allow to estimate the activation energy.

We have also obtained high-resolution spectra of inelastic light scattering spectra of the free-standing nitrocellulose film and at the interface of nitrocellulose/stainless steel, nitrocellulose/glass.

Observed experimental results demonstrate that the developed technique of high Spectral resolution inelastic light scattering has high potential for detection and study the nature of structural changes at the polymer/metal interfaces.

#### 9a. Plan for Following Year(s) (in the case the original work plan has been significantly changed)

As an example to demonstrate high spectral resolution of our system we performed measurements of high-resolution spectra of inelastic light scattering by longitudinal optical phonons in semi-insulating gallium phosphide crystals.

10a. References of papers and reports published.

1. Progress Report, Project # 2007p, For Q 1-2 (1 June – 30 November 2001).
2. Progress Report, Project # 2007p, For Q 3-4 (1 December 2002 – 31 May 2002).
3. Annual Report, Project # 2007p, For 1 year (1 June – 31 May 2002).
4. Progress Report, Project # 2007p, For Q 5-6 (1 June 2001 – 30 November 2002).
5. Annual Report, Project # 2007p, For 2 year (1 June 2001 – 31 May 2003).
6. Progress Report, Project # 2007p, For Q 9-10 (1 June 2003 – 30 November 2003).
7. Annual Report, Project # 2007p, For 3 year (1 June 2001 – 31 May 2003)

## II. FINAL REPORT

### 1. Title of the Project / Number of Final Report

Development of High Spectral Resolution Technique for Registration Quasielastic Light Scattering Spectra Including Rayleigh and Brillouin Scattering as a Diagnostic Tool in Materials Characterization

For 3 years

### 2. Contracting Institute

Ioffe Physico-Technical Institute  
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### 3. Participating Institutes

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### 5. Commencement Date, Duration

1 June 2001, for 3 years

6a. Brief description of the work plan: objective, expected results, technical approach

The main aim of the project is to carry out basic research on development high spectral resolution technique for registration quasielastic laser light scattering spectra. This technique is based on a use of nitrogen gas pressure scanned Fabry-Perot interferometer coupled to double grating monochromator. It also to detect quasielastic electronic light scattering spectra as well as Rayleigh, Brillouin and Raman scattering.

According to the Work Plan we have conducted the **Task 1:**

To develop and built a special metallic box with optical windows for Fabry-Perot interferometer, which will allow also to perform temperature and pressure stabilization.

### **Task 2**

To measure temperature dependent electrical conductivity (in the temperature range of 77-300 K) of metal/polymer/metal systems for determination mobility and concentration of carriers.

### **Task 3**

To develop high spectral resolution technique for registration quasielastic laser light scattering spectra. This technique will be based on use of a high stability nitrogen gas pressure-scanned Fabry-Perot interferometer coupled to a double grating monochromator.

### **Task 4**

To perform mapping high-resolution Raman scattering measurements in the temperature range of 77-300 K on epoxy/Al and teflon/Al interfaces to find quasielastic electronic scattering and the evolution of structural changes from the surface of polymer close to the polymer/Al interfaces.

### **Task 5**

To perform mapping Brillouin light scattering measurements on epoxy/Al and teflon/Al interfaces to find the evolution of the longitudinal modulus from the surface of polymer close to the polymer/Al interfaces.

### **Task 6**

To perform Rayleigh and quasielastic light scattering measurements on epoxy/Al, epoxy/Ag and teflon/Al interfaces to find the evolution of the relaxation time from the surface of polymer close to the polymer/Al interfaces.

## **7a. Technical progress during the 1 year of reference**

- compliance with tasks and milestones as described in the work plan

All works are on schedule.

- achievements of the first year

### **Task 1**

We have designed and built new special metallic box with two optical windows for the Fabry-Perot interferometer and fine valve. High optical-quality and plane windows were made by us from the quartz glass. This system will allow performing photoelectric recording of the spectra together with high temperature and pressure stabilization.

The main goal was achieving of the linear scanning of the system. The linear scanning of the Fabry-Perot interferogram is achieved by slow and linear change of the nitrogen gas pressure inside the metallic box by preliminary for-vacuum pumping and then slow filling of the system by the nitrogen gas using very fine valve. We used high spectral purity compressed nitrogen gas. Normal filling velocity can be varied from  $\sim 1 \times 10^{-2}$  to  $1 \times 10^{-4}$  l/s.

We were able to detect 7 orders of the interference fringes. Nonlinearity of the scanning for the first 3 orders was  $< 1\%$ . Usual time needed for scan of one order is  $\sim 5$  min. The spectral resolution of the system with the 632.8 nm excitation line of the He-Ne laser is  $0.06 \text{ cm}^{-1}$  for the free spectral range of  $2.50 \text{ cm}^{-1}$ . The accuracy of reading wavenumbers is  $0.005 \text{ cm}^{-1}$ .

Currently the system is ready for further applications. We will try to use this system for detecting quasielastic light scattering spectra as well as Rayleigh, Brillouin and Raman scattering. Also the use of the double grating monochromator allows drastically reducing of the amount of parasitic light, this problem for detection of the spectra of materials with high background scattering requires further study.

### **Task 2**

To study electrical properties of the polymer/metal interfaces we have prepared samples with commercial epoxy as a polymer and epoxy-aluminum joints. The interface structures were fabricated as following. The two-component homogeneous epoxy was inserted between aluminum and molybdenum covered subject glass plates with conducting electrodes.

The specimens with dimensions of 5x5 mm and thickness of approximately 0.1 mm were fabricated.

The electrodes were connected to high-impedance measurement voltmeter.

## **2. Results of measurements and discussions**

### **A) Current-voltage characteristics**

Current-voltage characteristics of the structures were measured by standard 4-point technique in the temperature range 77-300K with external

bias potential of 500 V. The current conductors were fabricated by aluminum and molybdenum films on the glass with thickness of 0.1 mm. As a potential conductors were used tungsten wires with diameter of 0.1 mm.

In measured range with current bias ponteial of  $U \leq 500$  V the current-voltage characteristics were independent on the nature of the metal and followed Ohm law  $I \propto U$  in the temperature range 80-300 K.

Typical current-voltage characteristics for two structures at  $T = 300$  K are show in the Fig. 1 (curve 1 for molybdenum-polymer- molybdenum structure and curve 2 for aluminum-polyme-aliminum structure.

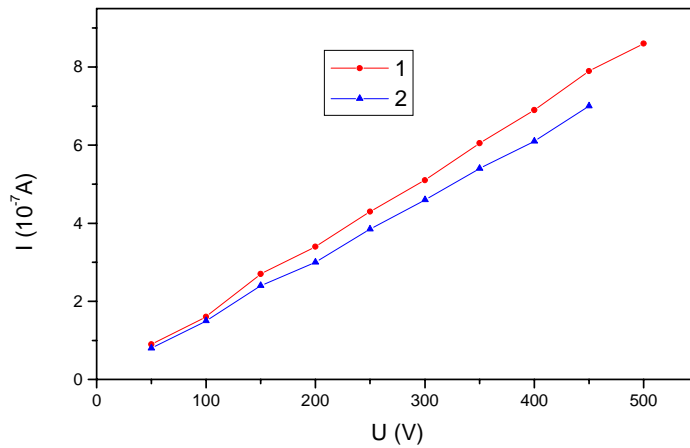


Fig.1. Current-voltage characteristics for the two structures metal-polymer-metal at  $T = 300$  K. Curve 1 for molybdenum-polymer- molybdenum structure and curve 2 for aluminum-polyme-aliminum structure.

### B) Stability of specific resistance

Metal-polymer-metal structures fabricated by above mentioned technique were characterized with the specific resistance of  $\rho \cong (2 - 5) \cdot 10^{10} \Omega$  (for different samples) at the room temperature.

We have not observed any degradation of the structures under thermocycling changes during 3-4 weeks.

Under the conditions of smooth reducing the temperature starting from  $T < 200$  K were revealed instability of the conductance that is connected with breakdown of close contacts at the metal-polymer interface due to the mismatch of thermal expansion coefficients of so different materials the metal and polymer.

### C) Hall effect

Hall effect for our polymer films was not observed due to small values of mobility in such highly resistive materials. If we take the known level of



the mobility  $\cong 10^{-2} - 10^{-4} \text{ cm}^2/\text{V}\cdot\text{s}$ . ( e.g., see J.Simon, J.J. Andre . Molecular Semiconductors. Eds J.M. Leh, Ch.W. Rees/ Springer-Verlag, N.-Y.,1988) then we obtain the following value of the carriers concentration  $\mu \cong 10^8 - 10^{10} \text{ cm}^{-3}$  at  $T = 300 \text{ K}$ .

#### D) Temperature dependence of the resistance

Our measurements of the resistance at different temperatures for the polymer-metal-polymer interfaces show that in the range of the stability of  $\rho$  in dependence of  $\lg \rho - 1/T$  the temperature dependence is aligning (see Fig. 2).

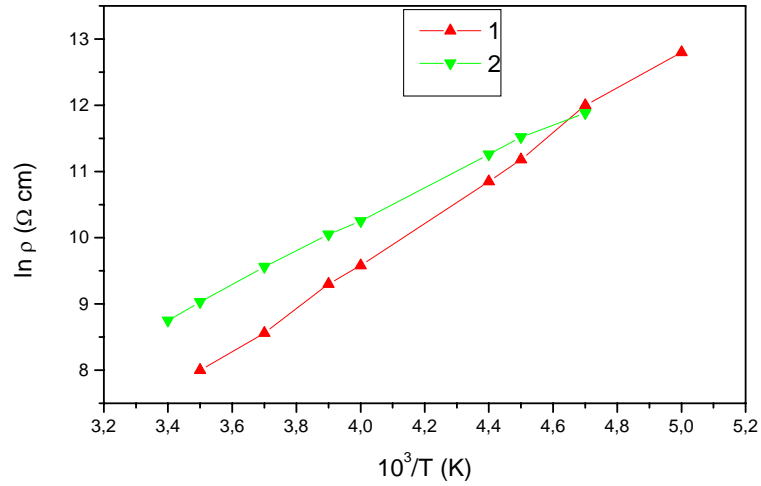


Fig. 2. Temperature dependence of specific resistance  $\rho(T)$  for the two structures metal-polymer-metal at  $T = 300 \text{ K}$ . Curve 1 for molybdenum-polymer- molybdenum structure and curve 2 for aluminum-polyme-aliminum structure.

Therefore, the temperature dependence can be described as

$$\rho = \rho_0 \exp (- E_\rho / 2 kT),$$

where  $E_\rho$  is the activation energy of unknown nature specific resistance. For the polymer-metal-polymer interfaces we obtain  $E_\rho = 2.2 - 3 \text{ eV}$ . In the frame of this models the activation energy  $E_\rho$  can be considered as difference between the conduction band and edge of filled valence band. So, the forbidden band gap equal to the energy required for creation free carrier pairs – electrons and holes.

Hence, our first study of electrical properties of the system with polymer-metal-polymer interfaces indicate the Ohm-law character of the conductivity for the polymer film inserted between metallic plates. Current-voltage characteristics measured in the wide temperature and potential ranges indicate that the temperature dependence of the conductivity is described by the exponential law, that allow to estimate the activation energy.

#### 8a. Current technical status

- on schedule, behind, ahead

All works are on schedule.

- refining next year schedule if necessary

We plan to study inelastic light scattering by longitudinal optical phonons in semi-insulating gallium phosphide crystals.

- recommendation for changes of the work plan, if necessary

Our measurements of the inelastic light scattering by longitudinal optical phonons in semi-insulating gallium phosphide crystals allowed to demonstrate high spectral resolution of our system.

#### 9a. Cooperation with foreign collaborators

- exchange of scientific material (information, computer codes and data, samples)

We present Progress Reports, for Q 1-2 (1 June – 30 November 2001) and for Q 3-4 (1 December 2002 – 31 May 2002).

- signature of protocols (with short description)
- research carried out jointly

#### **Task 3**

We have designed and built new special metallic box with the optical windows for the Fabry-Perot interferometer, which allows high temperature and pressure stabilization. We have measured the temperature dependent electrical conductivity (in the temperature range of 77-300 K) of metal/polymer/metal systems for determination mobility and concentration of carriers. In these system we plan to study the quasielastic light scattering.

- trips to/from foreign collaborators
- workshops, topical meetings organized by the project team
- joint attendance to international conferences

#### 10a. Problems encountered and suggestions to remedy

One the main question is the current problems with the detection of the low-level intensity of the both photoluminescence and electronic inelastic light scattering spectra. To solve this problem, we have needed acquire a new photomultiplier tube with the GaAs (Cs) photocathode for the visible spectral range (Hamamatsu R943-02).

Therefore it was need to reconsider our equipment and material summary and cut also early planned outside CIS travel program.

To detect the low-level intensity of the both photoluminescence and electronic inelastic light scattering spectra we acquired a new photomultiplier tube with the GaAs (Cs) photocathode for the visible spectral range (Hamamatsu R943-02). The required cooling system for the tube was developed and made by us.

#### 11a. Perspectives of future developments of the research/technology developed

Currently the system is ready for further applications. We will try to use this system for detecting quasielastic light scattering spectra as well as Rayleigh, Brillouin and Raman scattering. Also the use of the double grating monochromator allows drastically reducing of the amount of parasitic light, this problem for detection the spectra of materials with high background scattering requires further study.

### IIIa. ATTACHMENTS

#### I. Summary of personnel commitments for the 1 year

Category I – 186 days and Category II – 185 days.

#### II. Equipment acquired during the year.

No equipment was acquired.

#### 6b. Brief description of the work plan: objective, expected results, technical approach

##### **Task 3**

To develop high spectral resolution technique for registration quasielastic laser light scattering spectra. This technique will be based on use of a high stability nitrogen gas pressure-scanned Fabry-Perot interferometer coupled to a double grating monochromator.

##### **Task 4**

To perform mapping high-resolution Raman scattering measurements in the temperature range of 77-300 K on epoxy/Al and teflon/Al interfaces to find quasielastic electronic scattering and the evolution of structural changes from the surface of polymer close to the polymer/Al interfaces.

## 7b. Technical progress during the second year

### Task 3

We have developed a novel technique for detection high-resolution quasielastic light scattering spectra. The technique is based on made by us pressure scanned (nitrogen gas) Fabry-coupled to double grating monochromator. This system allows also performing photoelectric recording of the spectra together with the high temperature and pressure stabilizations.

The main aim of our project is to carry out basic research on development high spectral resolution technique for registration of quasielastic light scattering spectra including Rayleigh and Brillouin Scattering as a diagnostic tool in materials characterization. This technique is based on our development by use of pressure scanned Fabry-Perot interferometer coupled to double grating monochromator. The monochromator is used, in this case, as a spectral prefilter, which allow to select the spectral range need and to reduce the intensity of the parasitic spectra .

With the use of such a system we are able to perform photoelectric recording of the high-resolution spectra. Moreover we achieved high temperature and pressure stabilization. The spectral resolution of the system with the He-Ne laser 632.8 nm excitation line is  $0.06 \text{ cm}^{-1}$  for the free spectral range of  $2.50 \text{ cm}^{-1}$ .

Typical application of our system is shown on Fig. 1, which demonstrate instrumental contour with the high-resolution of the system as well as interferometric spectra of light scattering by optical phonons in semi-insulating GaP crystals in the temperature range 7 – 300 K. Two orders of the interference are shown.

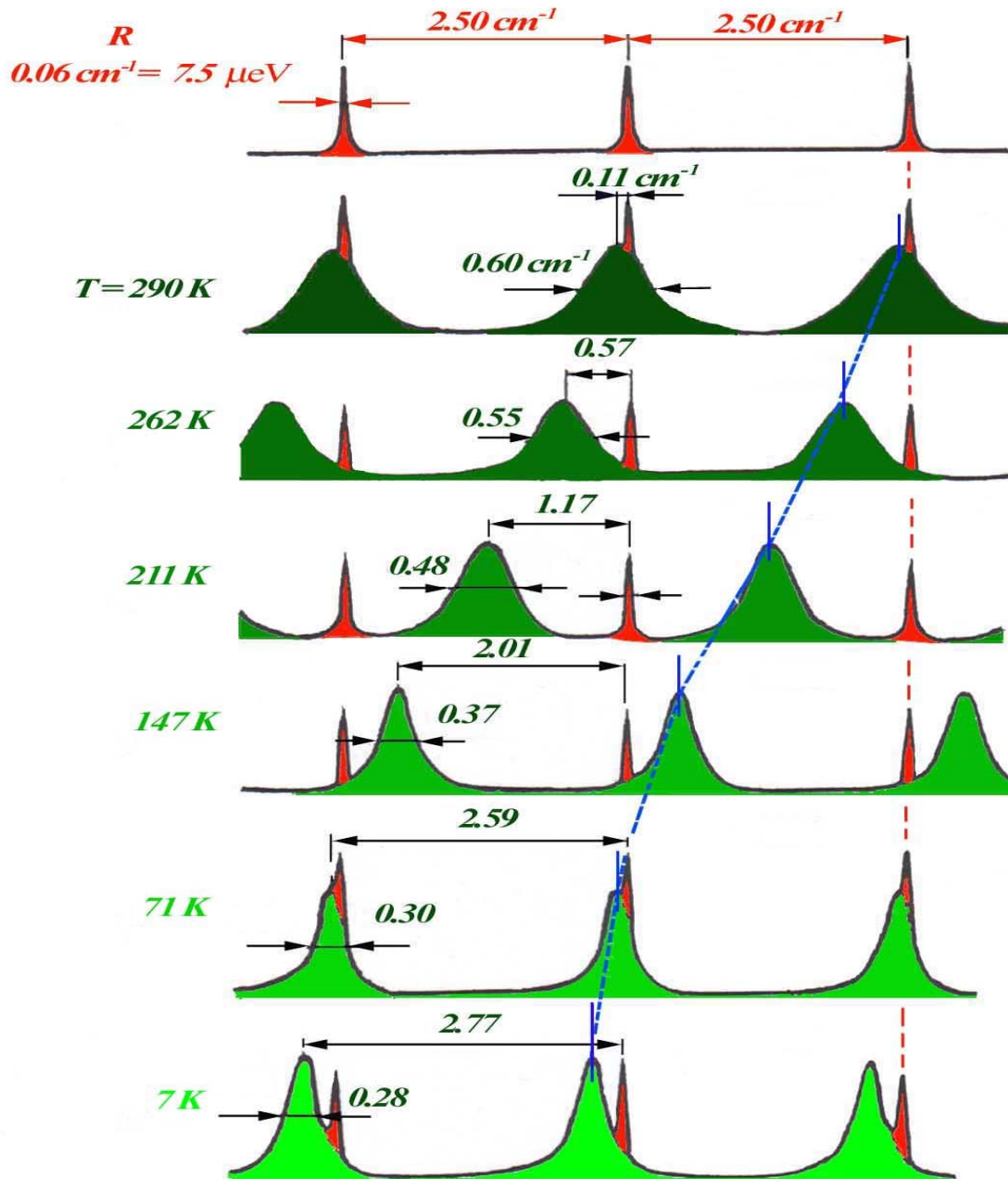


Fig. 3. Spectra of high-resolution light scattering by the longitudinal optical phonons in semi-insulating GaP crystals in the temperature range 7 – 290 K.

Currently the system is ready for further applications. We will try to use this system for detecting quasielastic light scattering spectra as well as Rayleigh, Brillouin and Raman scattering. Also the use of the double grating monochromator allows drastically reducing the amount of parasitic light, this problem for detection the spectra of materials with high background scattering requires further study.

Task 4.

For more adequate interpretation of our results instead of epoxy films we have used nitrocellulose.

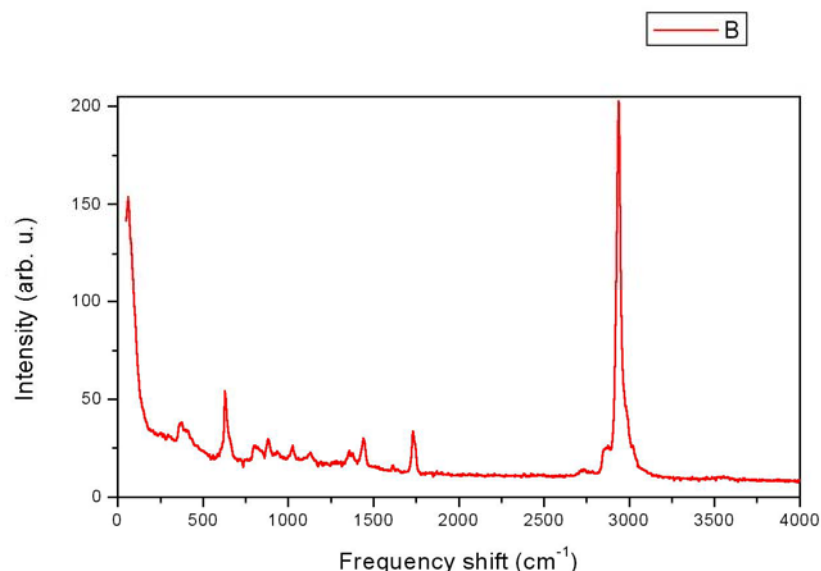


Fig. 4. Inelastic light scattering spectrum at the nitrocellulose/aluminum interface. The excitation was 514,532 nm line of the argon laser.

The typical spectrum of the inelastic light scattering at the nitrocellulose/aluminum interface is shown on Fig.5. For revealing the evolution of the structural changes starting from the surface until the interface nitrocellulose/metal we will perform measurements of inelastic light scattering spectra for the system nitrocellulose/stainless steel. These measurements will be performed under the same experimental conditions. As an example for demonstration of applications of high spectral resolution measurements of our system we also plan to study inelastic light scattering in doped gallium phosphide crystals.

- achievements of the past year

We have designed and built new special metallic box with two optical windows for the Fabry-Perot interferometer and fine valve. This system allows performing the photoelectric recording of the high-resolution spectra together with high temperature and pressure stabilization.

The main goal was achieving of the linear scanning of the system. The linear scanning of the Fabry-Perot interferogram is achieved by slow and linear change of the nitrogen gas pressure inside the metallic box by preliminary for-vacuum pumping and then slow filling of the system by the nitrogen gas using very fine valve. We used high spectral purity compressed nitrogen gas. Normal filling velocity can be varied from  $\sim 1 \times 10^{-2}$  to  $1 \times 10^{-4}$  l/s.

We were able to detect 7 orders of the interference fringes. Nonlinearity of the scanning for the first 3 orders was  $< 1\%$ . Usual time needed for scan of one order is  $\sim 5$  min. The spectral resolution of the system with the 632.8 nm excitation line of the He-Ne laser is  $0.06 \text{ cm}^{-1}$  for the free spectral range of  $2.50 \text{ cm}^{-1}$ . The accuracy of reading wavenumbers is  $0.005 \text{ cm}^{-1}$ .

Currently the system is ready for further applications. We will try to use this system for detecting quasielastic light scattering spectra as well as Rayleigh, Brillouin and Raman scattering. Also the use of the double grating monochromator allows drastically reducing of the amount of parasitic light, this problem for detection the spectra of materials with high background scattering requires further study.

#### 8b. Current technical status

- on schedule, behind, ahead

All works are on schedule.

- refining next year schedule if necessary

As an example for applications of our high-resolution system we plan to study inelastic light scattering by longitudinal optical phonons in doped gallium phosphide crystals as well as inelastic light scattering spectra at the nitrocellulose/stainless steel and nitrocellulose/glass interfaces.

- recommendation for changes of the work plan, if necessary

Our measurements of the inelastic light scattering by longitudinal optical phonons in gallium phosphide crystals with external perturbations (such as local deformations, low-level doping, etc.) will allow further demonstration of the high spectral resolution of our system.

#### 9b. Cooperation with foreign collaborators

- exchange of scientific material (information, computer codes and data, samples)

We present Progress Reports, for Q 5-6 (1 June – 30 November 2002) and for Q 7-8 (1 December 2002 – 31 May 2003) and 1 year annual report.

- signature of protocols (with short description)
- research carried out jointly

We have developed and demonstrated technique for measurements of high-resolution inelastic light scattering spectra. We have measured the temperature dependent electrical conductivity (in the temperature range of 77-300 K) of metal/polymer/metal systems for determination mobility and concentration of carriers. In these system we plan to study the quasielastic light scattering.

- trips to/from foreign collaborators
- workshops, topical meetings organized by the project team
- joint attendance to international conferences

#### 10b. Problems encountered and suggestions to remedy

One the main question is the current problem with the detection of the low-level intensity of the quasielastic light scattering spectra. Our preliminary results show high level of the parasitic background. We will try to conduct new measurements in the samples with high crystalline structure.

#### 11b. Perspectives of future developments of the research/technology developed

Currently the high spectral resolution system is ready for further useful applications. We will try to use this system for detecting quasielastic light scattering spectra as well as inelastic light scattering. Also the use of the double grating monochromator allows drastically reducing of the amount of parasitic light, this problem for detection the spectra of materials with high background scattering requires further study. Therefore we will try to study samples with high lattice perfection.

Attachment 1: Illustrations attached to the main text

Attachment 2: Other Information, supplements to the main text

Attachment 3: Abstracts of papers and reports published during the year of reference

1. Progress Report, Project # 2007p, For Q 5-6 (1 June 2001 – 30 November 2002).
2. Annual Report, Project # 2007p, For 2 year (1 June 2001 – 31 May 2003).

Attachment 4: Information on patents and property rights.

### IIIb. ATTACHMENTS

#### IIIb. Summary of personnel commitments for the 2 year

Category I – 186 days and Category II – 185 days.

#### III. Equipment acquired during the year.

No equipment was acquired.

#### 6c. Brief description of the work plan for year 3: objective, expected results, technical approach

According to the Work Plan we have conducted the **Task 5:**

To perform mapping Brillouin light scattering measurements on epoxy/Al and teflon/Al interfaces to find the evolution of the longitudinal modulus from the surface of polymer close to the polymer/Al interfaces.



## Task 6

To perform Rayleigh and quasielastic light scattering measurements on epoxy/Al, epoxy/Ag and teflon/Al interfaces to find the evolution of the relaxation time from the surface of polymer close to the polymer/Al interfaces.

### 7b. Technical progress during the 3 year

## Task 5

As we mentioned above we use the high-resolution inelastic light scattering technique developed by and have performed photoelectric recording of the high-resolution spectra at the nitrocellulose/aluminum interface, by the longitudinal optical phonons in the semi-insulating *si*-GaP crystals as well as in the *n*-GaP layers homoepitaxially grown on the bulk *n*-GaP substrate oriented along the crystallographic [100] direction.

At the same time the main problem with these measurements is still the high amount of parasitic (unshifted) light that masks detection of quasielastic light scattering spectra.

We will use high –precision measurements from free-standing nitrocellulose films to clarify the local structure at the surface of the nitrocellulose films and at the interface of nitrocellulose/aluminum.

Fig. 5a shows typical spectra for the two orders of interference that demonstrate high spectral resolution achieved. As an excitation line was used 632.817 nm line of the cw He-Ne laser. The spectral resolution of the system is  $0.06 \text{ cm}^{-1} = 7.5 \text{ } \mu\text{eV}$  with a free spectral range of  $2.50 \text{ cm}^{-1}$ .

For comparison Fig 5b shows interferometric high resolution inelastic light scattering spectra by the longitudinal LO( $\Gamma$ ) optical phonons in the semi-insulating *si*-GaP crystals with concentration of free carriers  $n \leq 10^{12} \text{ cm}^{-3}$  at room temperature. The full width at the half maximum is  $0.60 \text{ cm}^{-1}$  with a frequency shift  $0.11 \text{ cm}^{-1}$  while the full frequency of this phonon line is  $402.3 \text{ cm}^{-1}$ .

The spectra given in the Fig. 5c is received for the thin *n*-GaP layers homoepitaxially grown on the bulk *n*-GaP substrate oriented along the crystallographic [100] direction. In comparison to the spectra of the semi-insulating *si*-GaP crystals it is well-seen sharp bands broadened up to  $1.06 \text{ cm}^{-1}$  with an additional blue shift up to  $0.76 \text{ cm}^{-1}$ . At the same time no shift and broadening is observed for the transverse TO( $\Gamma$ ) optical phonons lines (not shown in the Fig. 5.)

Therefore the shift and broadening detected for the observed band of thin homoepitaxial *n*-GaP layers are not connected with local stress and inhomogeneity. They directly indicate to formation of the coupled states of the longitudinal LO( $\Gamma$ ) optical phonons with a plasma vibrations. Therefore this date can be used

determination of such a kinetic parameters as a concentration and mobility of free carriers.

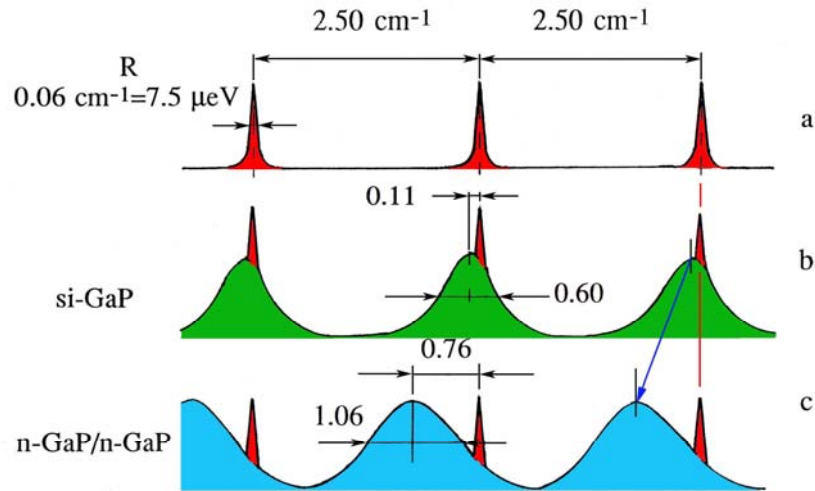


Fig.5. Typical spectra for the two orders of interference that demonstrate high spectral resolution achieved (a) and of inelastic light scattering spectra by the longitudinal  $LO(\Gamma)$  optical phonons in the semi-insulating *si*-GaP crystals (b) and for the thin *n*-GaP layers homoepitaxially grown on the bulk *n*-GaP substrate (c).

The excitation wavelength  $\lambda_i = 632.817$  nm.

The spectral resolution of the system is

$0.06 \text{ cm}^{-1} = 7.5 \text{ } \mu\text{eV}$  with a free spectral range of  $2.50 \text{ cm}^{-1}$ .

Note that realization of interferometric registration of inelastic light scattering spectra with high precision allows determination of crystalline perfection of the structures. But it is still remains the problems with the high amount of parasitic (unshifted) light that masks detection of quasielastic light scattering spectra.

## Task 6

We have conducted measurements of the inelastic light scattering measurements of free-standing nitrocellulose film and interface of nitrocellulose/stainless steel, nitrocellulose/glass and nitrocellulose/aluminum in the range of  $200 - 3500 \text{ cm}^{-1}$  by using the second harmonic cw  $532.19$  nm line of the  $\text{Nd}^{+3}$ -YAG laser as an excitation line. Mapping point by point measurements for the thin nitrocellulose/metal films indicated that the most intense lines of the nitrocellulose due to the intermolecular stretching vibrations of C-H and O-H bonds over the frequency range  $2600-3500 \text{ cm}^{-1}$  can be used to reveal local stress and homogeneity of the nitrocellulose/metal films.

Fig.6 shows typical inelastic light scattering spectra of free-standing nitrocellulose film and interface of nitrocellulose/stainless steel, nitrocellulose/glass and nitrocellulose/aluminum in the range of  $200 - 3500 \text{ cm}^{-1}$ .

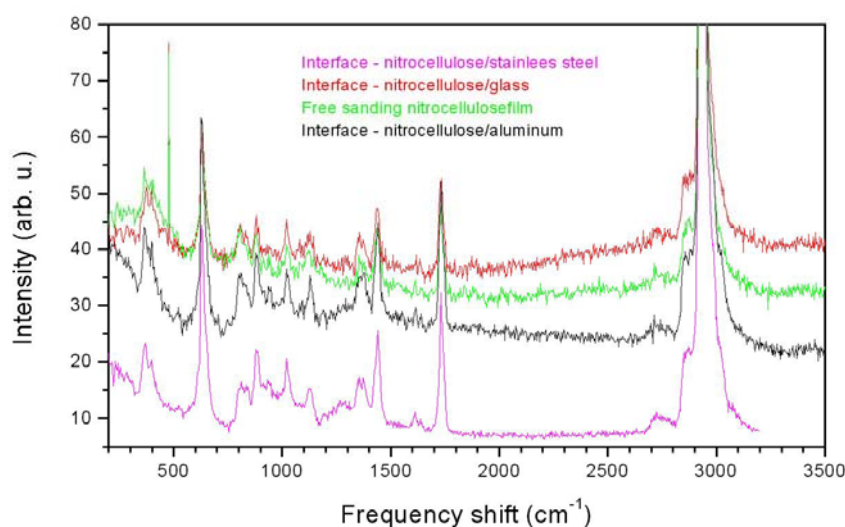


Fig. 6. Typical inelastic light scattering spectra of free-standing nitrocellulose film and interface of nitrocellulose/stainless steel, nitrocellulose/glass and nitrocellulose/aluminum films in the range of 200 – 3500  $\text{cm}^{-1}$ . The excitation wavelength  $\lambda_i = 532.19 \text{ nm}$ . The spectral resolution is better than 2  $\text{cm}^{-1}$ .

The spectra were obtained by using the second harmonic cw 532.19 nm line of the  $\text{Nd}^{+3}$ -YAG laser as an excitation line.

The vibrational spectra of nitrocellulose are very complex and requires more specific and additional extensive study. Therefore here we can give only approximate interpretations of the observed peaks.

Each of the spectrum as well as the spectra taken at different points for one sample are highly reproducible with all spectral parameters of the bands (frequency shifts, intensities and linewidths) indicating also the ability of technique developed for determination homogeneity of the nitrocellulose films at the different interfaces. For the spectra shown in Fig. 6 besides the observed changes in relative spectral characteristics of some bands there are also definite changes in background. All the spectra demonstrate predominant peaks due to the intermolecular stretching vibrations of C-H bonds over the frequency range of 2600-3300  $\text{cm}^{-1}$ .

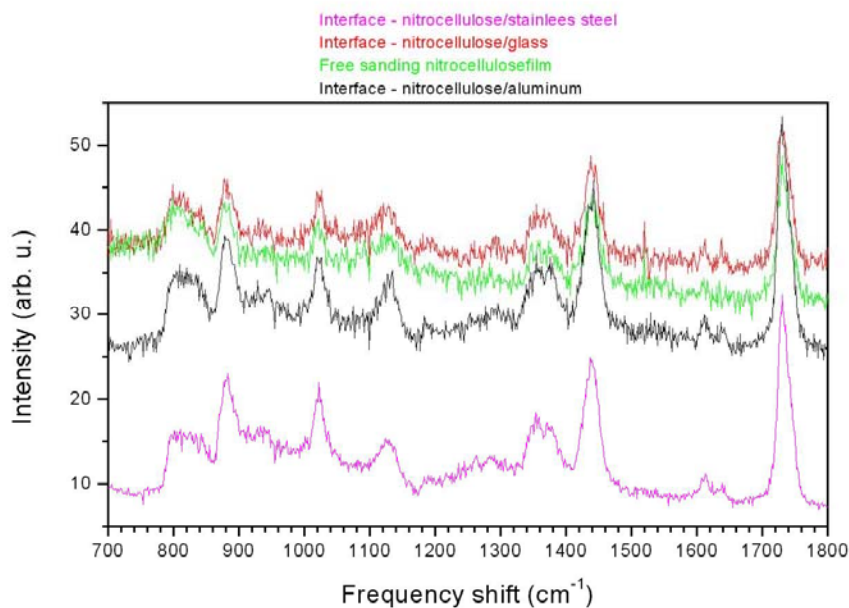


Fig. 7. Inelastic light scattering spectra of free-standing nitrocellulose film and interface of nitrocellulose/stainless steel, nitrocellulose/glass and nitrocellulose/aluminum films in the range of 200 –3500  $\text{cm}^{-1}$ .

Extended version of these spectra over the frequency range 700-1800  $\text{cm}^{-1}$  are shown in the Fig. 7 demonstrate more clearly different structural changes at the interfaces.

Not going into details the peak at around 1750  $\text{cm}^{-1}$  is due to C=O stretching mode while those in the range of 900 - 1500  $\text{cm}^{-1}$  can be attributed to  $\text{CH}_2$  bending vibrations and in the low frequency range to the bending modes involving CCC, COC, OCC and OCO internal vibrations.

In general, these spectra show approximately the same features. But the observation of narrow bands allows to study the details of nitrocellulose structure under condition of formation thin films at the different interfaces.

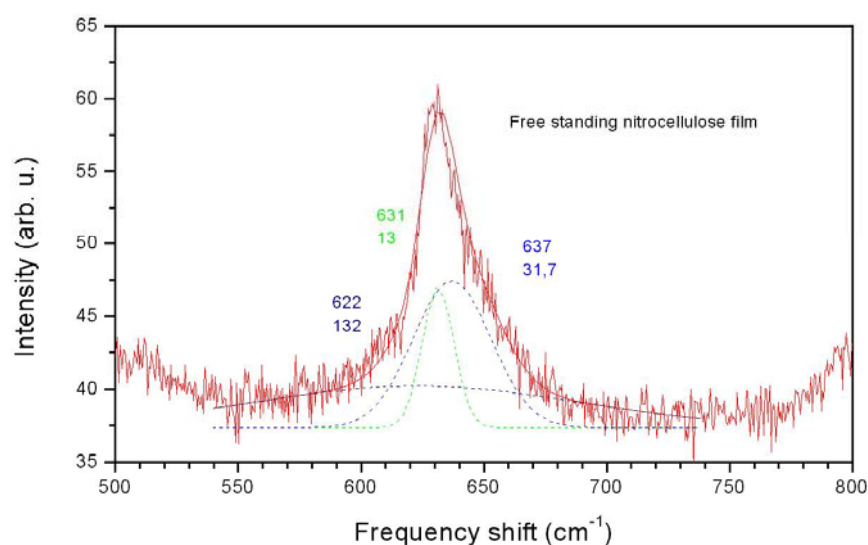


Fig. 8. Decomposition of inelastic light scattering spectra of free-standing nitrocellulose film around  $630\text{ cm}^{-1}$ . Figures indicate frequency shift and linewidth (FWHM full width at half of the maximum) for the each decomposed lines.

For example, in the extended spectra of the free-standing nitrocellulose film and interface of nitrocellulose/stainless steel, nitrocellulose/glass and nitrocellulose/aluminum in the range of  $500 - 800\text{ cm}^{-1}$  shown in Figs 8-10 this is clearly seen with obviously resolved decomposition structures in comparison with the spectrum of the free standing nitrocellulose in the Fig. 8.

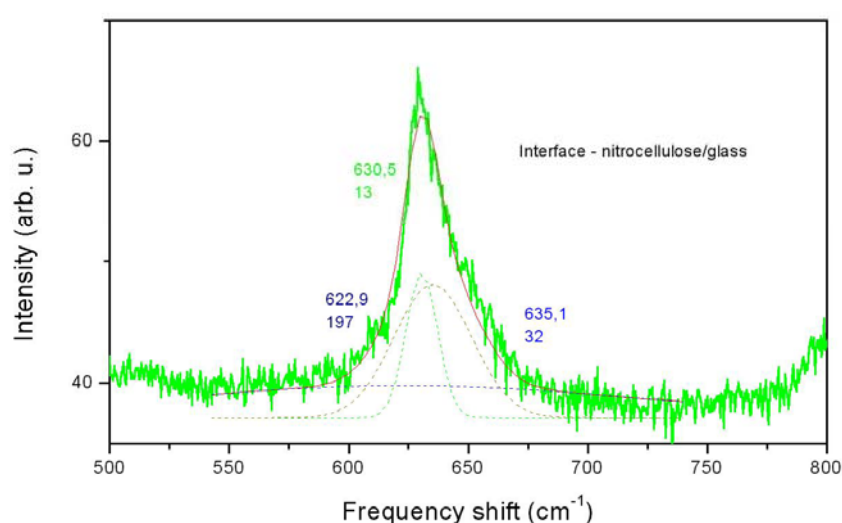


Fig. 9. Decomposition of inelastic light scattering spectra of the interface nitrocellulose/glass around  $630\text{ cm}^{-1}$ . Figures indicate

frequency shift and linewidth (FWHM full width at half of the maximum) for the each decomposed lines.

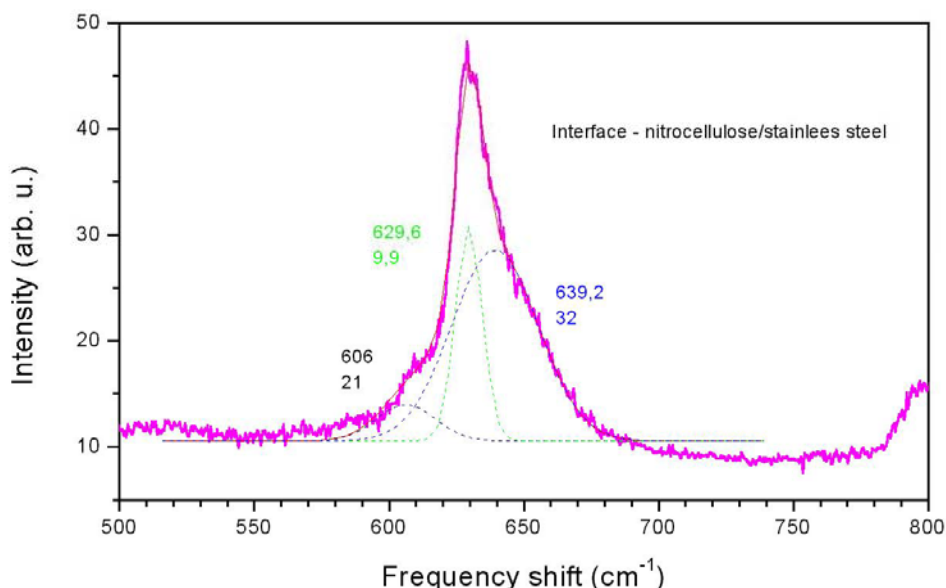


Fig. 10. Decomposition of inelastic light scattering spectra of the interface nitrocellulose/stainless steel around  $630\text{ cm}^{-1}$ . Figures indicate frequency shift and linewidth (FWHM full width at half of the maximum) for the each decomposed lines.

Observed experimental results demonstrate that the developed technique of high resolution inelastic light scattering has high potential for detection and study the nature of structural changes at the polymer/metal interfaces.

With this project we were able to stabilize our research activity and develop a high spectral resolution technique. The system allows precise registration of inelastic and quasielastic light scattering spectra and we demonstrated the possibility of the detection the high-resolution spectra for a number of materials. It will be attractive to continue our fundamental research activity in this field that opens up completely new comprehensive approaches for better understanding basic problems of formation structural changes at the polymer/metal interfaces.

#### 8c. Current technical status

- on schedule, behind, ahead

All works were on schedule.

#### 9c. Cooperation with foreign collaborators

- exchange of scientific material (information, computer codes and data, samples)

We present:

1. Progress Report, Project # 2007p, For Q 1-2 (1 June – 30 November 2001).
  2. Progress Report, Project # 2007p, For Q 3-4 (1 December 2002 – 31 May 2002).
  3. Annual Report, Project # 2007p, For 1 year (1 June – 31 May 2002).
  4. Progress Report, Project # 2007p, For Q 5-6 (1 June 2001 – 30 November 2002).
  5. Annual Report, Project # 2007p, For 2 year (1 June 2001 – 31 May 2003).
  6. Progress Report, Project # 2007p, For Q 9-10 (1 June 2003 – 30 November 2003).
  7. Annual Report, Project # 2007p, For 3 year (1 June 2001 – 31 May 2003).
- signature of protocols (with short description)
  - research carried out jointly
  - trips to/from foreign collaborators
  - workshops, topical meetings organized by the project team
  - joint attendance to international conferences

#### 11. Problems encountered and suggestions to remedy

#### 12. Perspectives of future developments of the research/technology developed

Currently the developed high spectral resolution optical system for the detection highly precision inelastic light scattering spectra in the crystals, polymers, water solutions, biomedical samples and etc., is ready for further useful investigations and applications.

Attachment 1: Illustrations attached to the main text

Attachment 2: Other Information, supplements to the main text

Attachment 3: Abstracts of papers and reports published during the year of reference

1. Progress Report, Project # 2007p, For Q 1-2 (1 June – 30 November 2001).
2. Progress Report, Project # 2007p, For Q 3-4 (1 December 2002 – 31 May 2002).
3. Annual Report, Project # 2007p, For 1 year (1 June – 31 May 2002).
4. Progress Report, Project # 2007p, For Q 5-6 (1 June 2001 – 30 November 2002).
5. Annual Report, Project # 2007p, For 2 year (1 June 2001 – 31 May 2003).

6. Progress Report, Project # 2007p, For Q 9-10 (1 June 2003 – 30 November 2003).
7. Annual Report, Project # 2007p, For 3 year (1 June 2001 – 31 May 2003).

Attachment 4: Information on patents and property rights.

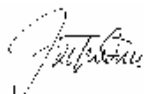
### **IIIc. ATTACHMENTS**

#### **I. Summary of personnel commitments for the 3 year**

Category I – 407 days, Category II – 376 days and Category IV -22 days.

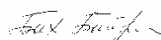
### **IV. SIGNATURES**

Deputy Director  
Ioffe Physico-Technical Institute



A. G. Zabrodskiy

Project Manager



B. H. Bairamov

05/31/04